Irradiation of Nd-Fe-B Permanent Magnets with APS Bending Magnet X-rays and ⁶⁰Co γ-rays

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Introduction

The Advanced Photon Source (APS), as well as other third-generation synchrotron light sources, uses permanent magnets in the insertion devices to produce x-rays for scientific research [1,2]. When placed in a high-energy storage ring, these permanent magnets are subjected to irradiation from synchrotron radiation, high-energy bremsstrahlung, and bremsstrahlung-produced photoneutrons. Previous investigations have exhibited varying degrees of degradation in the intensity of magnetization of these magnets [3] due to irradiation from electron beams [4,5,6,7,8], 60 Co γ -rays [5,7,9], and high-energy neutrons [3,10,11]. The APS specifically uses Nd-Fe-B permanent magnets in their insertion devices [2]. Although no detectable radiation-induced demagnetization has been observed in the APS insertion devices so far [12], partial demagnetization has been observed in at least one insertion device at the European Synchrotron Radiation Facility (ESRF) [4,6], where Nd-Fe-B permanent magnets are also used.

A growing concern for the APS insertion devices as well as the insertion devices of next-generation light sources, such as the Free Electron Laser (FEL) [13,14] where permanent magnets will also be used, resulted from the partial demagnetization observed at the ESRF. Next-generation light sources will by far exceed the third-generation sources in terms of brightness, coherence, beam power, and energy [14]. The brightness of radiation produced in such sources is expected to be several orders of magnitude higher than current, third-generation sources [15]. This increase in brightness from the enhanced synchrotron radiation would result in a greater absorbed dose to the insertion devices from synchrotron radiation scattering.

Bremsstrahlung radiation will also be several orders of magnitude higher for future light sources and is expected to increase the absorbed doses to the insertion devices. The high power output of these next generation sources is expected to degrade the vacuum of the long straight sections many orders of magnitude. This vacuum degradation due to radiation desorption will increase the absorbed dose received by the insertion devices due to increased gas bremsstrahlung, as gas bremsstrahlung from the straight section increases linearly with pressure [16]. The longer straight section of approximately 50 meters will also account for an increase in bremsstrahlung. Larger beam bunches required for the single-pass operation of linear accelerators used in next-generation sources again will contribute to an increase in bremsstrahlung, as measurements at the APS showed that bremsstrahlung increases as the square of the transverse bunch profiles [16]. Smaller vacuum chambers proposed for use in the next-generation sources may also contribute to the absorbed doses due to bremsstrahlung depending on the beam location inside the chamber. Bremsstrahlung-produced neutrons, resulting from interactions with high-Z materials in the beamlines [17], will also factor into the absorbed doses received by the insertion devices of current, as well as next-generation sources.

These increased radiation concerns for the insertion devices to be used in future light sources, as well as lifetime concerns for the devices used in the present third-generation sources, prompted an investigation into the irradiation effects displayed in the permanent magnets used in insertion devices. A systematic irradiation of sample permanent magnets to very high radiation doses was carried out to determine the degree of degradation in the intensity of magnetization of these magnets using x-rays and γ-rays. Through a collaboration with the National Institute of Standards and Technology (NIST), four sample magnets were irradiated with 60Co γ-rays to an absorbed dose of approximately 700 Mrad. Similarly, with the cooperation of the Complex Material Consortium Collaborative Access Team (CMC-CAT), sample magnets were systematically irradiated with x-rays at APS beamline 9 BM to an approximate absorbed dose of 280 Mrad. The irradiation of Nd-Fe-B permanent magnets, similar to those used in the APS insertion devices, provides important information concerning the radiationinduced demagnetization of the insertion devices due to x-rays and γ -rays. This information is important to the designers of the insertion devices of the future generation of high-power light sources, as well as to users of the current synchrotron facilities.

Radiation-Induced Demagnetization of Permanent Magnets

Previous investigations into the radiation-induced demagnetization of permanent magnets similar to those used in the APS insertion devices produced varying results. Okuda et al. [5] performed a γ -ray irradiation of Nd-Fe-B sample magnets in a 60 Co irradiation facility at the Institute of Scientific and Industrial Research (ISIR) at Osaka University. The sample magnets were irradiated in the γ -ray source at a temperature of 20°C for a period of 459 hours [5]. The total absorbed dose to the magnets was determined using Celium dosimeters. The magnetic flux loss for the 60 Co γ -ray irradiation of Nd-Fe-B permanent magnets at an absorbed dose rate of 610 krad/h to an absorbed dose of 280 Mrad was below 0.5% [5].

Results cited in Luna et al. [7] stated a -0.00% remanence loss for the γ -ray irradiation of Nd-Fe-B permanent magnets. A sample of Nd₂Fe₁₄B alloy was placed in a ⁶⁰Co tank and irradiated to a maximum dose of 69 Mrad. "Initial" and "final" irradiation remanence readings were measured using a hand-held Hall probe gauss meter [7]. Boockmann et al. [9] measured the effect of γ -ray irradiation from a ⁶⁰Co source on the magnetic properties of sintered (Nd,Dy)₁₅Fe₇₇B₈ magnets. Up to an accumulated dose of about 50 Mrad neither the remanent polarization nor the intrinsic coercivity were significantly affected. The remanent polarization of every magnet was measured along with the demagnetization curve [9].

Numerous studies involving the irradiation effects of direct electron beams on Nd-Fe-B permanent magnet samples have been conducted. Direct electron beam irradiation yields useful results for situations when the particle beam is lost directly on the magnet. If the particle beam, or electron beam were missteered, or lost, directly on the magnet, an electromagnetic shower would be produced. The subsequent irradiation of the magnet would be the direct result of the electromagnetic shower produced, consisting of γ -rays, electrons, protons, and photoneutrons. An electron dose of 260 Mrad was used to irradiate Nd-Fe-B sample magnets with the 17-MeV L-band rf linear accelerator at the ISIR at Osaka University [5,18]. The magnetic flux of the sample magnets was measured using an open coil, evaluating the total magnetic flux of the sample [5]. The flux loss at an absorbed dose of 260 Mrad was found to be 9% [5,12], with an increase in flux loss for higher doses [5].

A similar study using an 82-MeV electron beam was performed at the linear accelerator of Lawrence Livermore National Laboratory [7]. Magnetic measurements compared the open circuit remanence of each sample magnet before and after irradiation using an integrated fluxmeter [7]. Two sample magnets were irradiated with a direct 82-MeV electron beam to a total dose of 36 krad [7]. The Nd₂Fe₁₄B sample showed a 1.5% remanence loss that was determined statistically significant. Exposure to bremsstrahlung radiation from an 85-MeV electron beam to a total dose of 450 Mrad resulted in a 14% remanence loss [7,12]. A second Nd-Fe-B sample, from a different manufacturer, displayed a 2% remanence loss after bremsstrahlung irradiation to a total dose of 1370 Mrad, suggesting that magnet composition from different manufacturing processes may have an effect on the rate of remanence loss [7,12].

The partial demagnetization of the two insertion devices at the ESRF, from missteering of the electron beam onto the vacuum chamber wall of the device, resulted in severe degradation of magnetic properties [8]. The estimated absorbed dose to one of the damaged devices was 6.7 Mrad to the first upper magnet, and 5.1 Mrad to the first lower magnet [12]. This dose resulted in a peak field loss at the upstream end of the device of approximately 8% [12], with demagnetization over one third of the length of the device [6]. This observed demagnetization at the ESRF prompted a subsequent irradiation program to test the sensitivity of various types of permanent magnets to electron beam irradiation. Three different types of Nd-Fe-B samples, along with two Samarium-Cobalt samples, were irradiated with a 180-MeV electron beam from the ESRF linear accelerator [4,8]. The samples were irradiated for one hour to an estimated dose of 70 Mrad [4,8]. All of the Nd-Fe-B samples showed partial demagnetization, with greater demagnetization observed in the samples with higher remanence and lower coercivity [4].

The ESRF partial demagnetization also prompted a similar study at HASYLAB that focused on possible radiation damage to permanent magnet insertion devices during routine operation of the storage ring. In this case the permanent magnets had been subjected to a realistic irradiation environment within the storage ring, where the particle beam usually is not lost directly on the magnets. This particular study focused primarily on the x-ray irradiation of the permanent magnets over time, rather than irradiation from the electromagnetic shower produced from direct electron beam irradiation. However a pure x-ray irradiation was not conducted specifically for this study. Magnetic measurements on an x-ray wiggler at HASYLAB were repeated in 1994 and compared to measurements taken during acceptance tests done prior to installation in 1991 [8]. The total dose was measured on the surface of the upstream end of the magnetic structure at different horizontal positions using thermoluminescence dosimeters [8]. The total estimated dose during the two-year period, corresponding to a total stored current of 214 Amp-hours, was 7.2 Mrad directly above the beam, and 3.3 Mrad and 12 Mrad at positions displaced horizontally by 40 mm [8,12]. Extensive magnetic measurements included field mapping using Hall probes, field integral measurement using short coils and Hall probes, determination of normal and skew integrated multipole components, as well as gap dependence of field integrals [8]. As a result of these extensive measurement procedures, it was determined that radiation-induced effects could not be detected within the accuracy of the measurements.

Resources Required for Irradiation

Irradiation of the Nd-Fe-B permanent magnets required resources encompassed in three distinct disciplines including high dose dosimetry, magnetic measurements, and temperature measurements. In order to measure x-ray irradiation doses greater than 10⁶ rad received by the sample permanent magnets, a reliable photon high-dose dosimetry technique was required, which was not readily available at the APS at the time. Through a collaboration with NIST, a technique using radiachromic films was considered, tested, and calibrated at the APS [19]. Radiachromic films are nylon-based aminotriphenyl

methane dye derivatives [20,21]. Upon exposure to ultraviolet light or ionizing radiation, the films undergo radiation-induced coloration by photoionization [20,22]. The radiation-induced photochemical reaction is displayed in Figure 1. This reaction results in a change from a clear or colorless state to a deep blue-colored state, which occurs gradually as a direct function of the radiation exposure received [20,23]. The change in the color intensity, or optical density, is measured using an optical reader, or a simple spectrophotometer.

The radiachromic films used for the magnet irradiation were FWT-60 films procured from Far West Technology of Goleta, California. These films have a linear response to ionizing radiation over a dose range of approximately 0.1 Mrad to 10 Mrad [20,23,24]. The films also have an equivalent response to x-rays, γ -rays, and electrons from ultraviolet energies up to approximately 1 MeV [25,26,27]. Radiachromic films display a constant response for exposures in air and vacuum, as well as in the presence of ozone [20,21,28]. Absorbed doses measured are relatively insensitive up to an ambient temperature of 50°C [23].

Through a collaboration, the radiachromic films were calibrated at the NIST standard gamma irradiation facility, GC232. The irradiation dose rate was 936.3 krad/hr, and the films were calibrated up to 10 Mrad. During the calibration the relative humidity was maintained at 44%. The individual films were placed inside paper detector envelopes and then sealed in aluminized Mylar pouches to protect them from ultraviolet exposure. For the calibration irradiation, the pouches were placed between two 5.0-mm-thick blocks of polystyrene to simulate tissue absorption. They were then placed upright on a polystyrene pedestal. The temperature during irradiation was controlled with forced air blown down on the samples and measured every sixty seconds. The calibration curve for the FWT-60 radiachromic films up to a dose of approximately 20 Mrad is given in Figure 2.

Measurement of the degree of degradation in the intensity of the magnetization of the Nd-Fe-B permanent magnets as a result of x-ray and γ-ray irradiation required a magnetic measurement system. This system was comprised of a Helmholtz coil and a fluxmeter. An eight-inch Helmholtz coil, Model HC-8, and a Model 2130 Fluxmeter were procured from Magnetic Instrumentation Inc., of Indianapolis, Indiana. Helmholtz coils were found to be ideal for the measurement of rare earth (Samarium Cobalt, Neodymium) and hard ferrite permanent magnets [29,30]. The Helmholtz coil measures the magnet sample as a single magnetic moment provided that the magnet sample's longest dimension is less than one third of the diameter of the coil [29,30]. By definition the magnetic moment per unit volume is the intrinsic magnetization of the sample. When the Helmholtz coil is used in conjunction with the Model 2130 Fluxmeter, and given the volume of the permanent magnet sample, the intrinsic open circuit operating B, or measure of the intrinsic remnant induction in a magnet sample is given in units of Bdi-Tesla [29]. The units of Bdi-Tesla are based on coil and magnet parameters; therefore, the Helmholtz coil constant and the coil resistance as well as the volume of the magnet sample must be known and entered into the coil parameter and magnet volume menus of the fluxmeter prior to magnet measurement. A list of the sample magnet and coil parameters for the magnetic

measurements used in conjunction with irradiation of the Nd-Fe-B sample magnets is provided in Table 1.

To determine the precision of the fluxmeter in terms of the measurement of the intrinsic remnant induction of the sample magnets, 100 measurements were taken for each of four sample magnets. The standard deviation was then calculated for each of the four data sets. In all cases the standard deviation was approximately $2x10^{-3}$. Assuming a normal distribution, 99% of the sample measurements will fall within three standard deviations (3 σ) of the sample mean. Using $2x10^{-3}$ as the average standard deviation (σ), three standard deviations give a range of \pm 0.006. Therefore, measured changes in the intrinsic remnant induction greater than 0.006 will be detected using this fluxmeter.

The other resources required for irradiation of the Nd-Fe-B permanent magnets are in the area of temperature measurement. Two aspects of the irradiation required some type of temperature control and measurement, including the radiachromic films and the sample magnets themselves. As stated before, the radiachromic films have no temperature dependence up to 50°C [2]. Because the films were placed directly on the sample magnets during the x-ray irradiation, the sample magnets had to be kept below 50°C.

The main disadvantage of Nd-Fe-B permanent magnets is their low Curie temperature. This leads to increased sensitivity to thermal demagnetization and limits their maximum temperature range [8,31]. The maximum use temperature for commercially available permanent magnets is typically less than 120° C [32]. The APS insertion device permanent magnets were all stabilized before the assembly of the devices so that temperatures up to 60° C would not cause any temperature-induced demagnetization [12]. In order to hinder temperature-induced demagnetization so as not to interfere with the radiation-induced demagnetization results, the temperature had to be kept below 60° C for the γ -ray irradiation and below 50° C for the x-ray irradiation due to the temperature constraint of the radiachromic films.

Viewdac (Revision 2.20), a control program from Keithley Instruments, Inc. was installed on a PC for the sole purpose of providing a temperature measurement system to use during the x-ray irradiation of the sample permanent magnets. The software was loaded and a specific program was written that allowed six thermocouples to monitor six separate temperatures during the irradiation. The thermocouples were CHROMEGATM/ALOMEGATM, 0.10" diameter / 36" length, Teflon-insulted thermocouples procured from OMEGA Engineering, Inc. of Stamford, CT. This temperature measurement system encompassed the resources needed for measurements that were required for temperature control during the x-ray irradiation of the sample magnets. The temperature during the γ -ray irradiation was held constant at 30°C by forced air cooling of the irradiation setup with house air at 20 psi; therefore temperature-induced demagnetization of the sample permanent magnets was not an issue.

Following the irradiation, statistical inference and a pooled t-test [33] were used to determine if a difference existed between the intrinsic remnant induction averages taken prior to irradiation and the average measurements taken after irradiation. Because of the

small sample size, n=5 readings taken for each magnet, the populations were assumed to be normally distributed, and the hypothesis test was based on confidence intervals and the *t*-distribution [33]. The difference in means, μ_1 = preirradiation mean and μ_2 = postirradiation mean, was determined by combining the two sample variances, s_1^2 and s_2^2 , to form an estimator of the variance of the normal distribution σ^2 . This estimator, called a pooled estimator of σ^2 , is denoted by S_p^2 .

A hypothesis test was formed using a null hypothesis H_0 and an alternative hypothesis H_1 . In this case we wished to test whether or not the means were equal, or H_0 : $\mu_1 = \mu_2$ and H_1 : $\mu_1 \neq \mu_2$. The purpose of the test is to use a test statistic t_0 that will support either the acceptance or rejection of the null hypothesis H_0 . The pooled estimator of σ^2 was found using sample sizes $n_1 = n_2 = 5$, s_1^2 , and s_2^2 set equal to the sample variances, and the following equation:

$$S_p^2 = [(n_1-1)*s_1^2 + [(n_2-1)*s_2^2]/[n_1+n_2-2].$$

The confidence interval was chosen, in this case the 99% confidence interval with $\alpha = 0.01$. This was used to determine the critical value $t_{\rm cr}$ that, when compared to the test statistic $t_{\rm o}$, supports either the acceptance or rejection of the null hypothesis. Values for $t_{\rm cr}$ are taken from statistical tables listing the percentage points of the t-distribution [33].

The test statistic was computed using the sample averages of the intrinsic remnant induction, the pooled estimator, sample sizes n_1 and n_2 , and the following equation:

$$t_0 = [\mu_1 - \mu_2 - 0] / [S_p * sqrt((1/n_1) + (1/n_2))].$$

The test statistic has a t-distribution with $(n_1 + n_2 - 2)$ degrees of freedom under H_0 : $\mu_1 = \mu_2$. If $t_0 > t_{cr}$ or if $t_0 < -t_{cr}$, the null hypothesis H_0 is rejected providing evidence at the 0.01 significance level, or the 99% confidence interval, that the two means differ.

γ-ray Irradiation

A systematic γ -ray irradiation of the Nd-Fe-B permanent magnets was done through a collaboration with the NIST Ionizing Radiation Division. The NIST standard gamma irradiation facility, Gammacell® GC232, a Cobalt-60 irradiation facility, was used to irradiate the magnets. Four sample magnets along with two control magnets were sent back and forth to NIST for a total of six irradiations. For each of the first five irradiations, the four sample magnets were irradiated in absorbed dose increments of 100 Mrad. The magnets were irradiated in an absorbed dose increment of 200 Mrad for the sixth irradiation. The six γ -ray irradiations resulted in a total absorbed dose to the sample magnets of approximately 700 Mrad.

As stated previously, the temperature during the γ -ray irradiation was held constant within the irradiation facility, which helped to control any temperature-induced demagnetization of the sample magnets. The temperature was kept at approximately 30°C

with air blown down onto the samples at 20 psi. This was well below the 60°C temperature requirement for the APS magnets. The high-dose dosimetry component of the irradiation was also satisfied by the NIST irradiation facility through the calibrated Gammacell®. Calibrated radiachromic films, in pouches, were placed between the magnets for a three-hour irradiation to verify an absorbed dose rate of 708.6 krad/hr.

Prior to sending the magnets to NIST for irradiation, intrinsic remnant induction readings were taken at the APS for each magnet. Four sample magnets, along with two control magnets were read using the magnetic measurement system previously described. Five measurements of the intrinsic remnant induction were taken for each sample magnet, as well as for the two control magnets. The average of these measurements served as initial readings to be compared to measurements taken after irradiation. All six magnets were wrapped in cardboard, approximately 3-mm thick, to reinforce safe handling. The four sample magnets were then stacked upright and wrapped in another layer of cardboard. They were then shipped along with the two control magnets to NIST for irradiation.

The magnet configuration described above was left in place during irradiation of the four sample magnets. Once the stacked arrangement of magnets was placed inside the center of the irradiation chamber, two rings of cardboard were fit onto each end to fill the chamber diameter and keep the magnets centered and upright. The Gammacell® itself consists of an annular source permanently enclosed within lead shielding, a cylindrical drawer, and a drive mechanism to move the drawer up and down along the vertical source center line [34]. A chamber in the drawer is used to carry the samples from outside the shield to inside the source for irradiation. The ⁶⁰Co source cage contains 48 "pencil" source positions. These tubular "pencil" sources each contain seven ⁶⁰Co slugs that are completely sealed in by welded end caps [34]. They are placed in an annular formation on an 8.32-inch pitch circle diameter around the source cage. A diagram of the source cage and arrangement of the "pencil" sources is shown in Figure 3.

After irradiation the sample magnets were packaged along with the control magnets and sent back to the APS. Upon arrival, five measurements of the intrinsic remnant induction were taken for each sample magnet as well as for the two control magnets. An average was taken for each set of measurements. These "final" averages were then compared to the "initial" averages taken prior to irradiation, using the pooled t-test previously described, to determine if any measurable radiation-induced demagnetization had occurred.

Following statistical analysis of the "initial" and "final" readings, "initial" intrinsic remnant induction readings were again taken at the APS for each of the four sample magnets as well as the two control magnets. The magnets were then sent back to NIST for the next irradiation in the cycle. This process continued for a total of six irradiations resulting in a total absorbed dose to the sample permanent magnets of approximately 7×10^8 rad.

Results and Analysis of γ-ray Irradiation

Intrinsic remnant induction measurements for each magnet prior to γ -ray irradiation compared to measurements following irradiation revealed small but not insignificant levels of variation. Tables 2-7 show the results as a percent change for each irradiation step. Figure 4 shows the same results, but provides a graphical format. From the tables, a 0.1% - 0.2% variation occurs at each irradiation step, but is not a cumulative effect. Because high accuracy is required in the magnetic fields of insertion devices, changes of this magnitude may cause diminished performance. Typically a 0.5% change in the magnetic field of insertion devices cannot be tolerated [5]. However, the 0.1% - 0.2% level of variation observed in this case is right at the edge of the precision capabilities of the fluxmeter, and is observed in the control magnets as well. Therefore, the variations are not statistically significant and may be attributable to the precision level of the instrument rather than radiation-induced demagnetization.

The change at each step in the γ -ray irradiation process was never more than 0.1% - 0.2% as compared to the measurements taken prior to the first irradiation. In other words, there was not a cumulative effect. If the change in the intrinsic remnant induction were the result of radiation-induced demagnetization, greater changes with increasing irradiation levels would be expected. The precision level of the instrument accounting for the observed variations may explain the absence of this effect, but it may also be due to the "stepwise" irradiation procedure. An annealing effect may have been taking place between irradiations, resulting in a 0.1% - 0.2% total change after each irradiation rather than a 0.1% - 0.2% change for each irradiation added cumulatively over all six irradiations.

The pooled t-test, described previously, was used to compare the preirradiation means of the intrinsic remnant induction measurements to the means found at each step in the irradiation process. Almost every comparison done using the test revealed evidence of differences between the means. However, the 0.1% - 0.2% level of variation is right at the edge of the precision capabilities of the fluxmeter, so although there may be differences between the means, the variations may be attributable to the precision level of the instrument rather than radiation-induced demagnetization.

The results of this study agree with previous work [5,7,9] that shows the effect of γ -ray irradiation on the radiation-induced demagnetization of Nd-Fe-B permanent magnets is small, but not insignificant if the changes can be determined statistically significant. However, due to the precision capabilities of the fluxmeter, the 0.1% - 0.2% level of variation observed in this study cannot be attributed to radiation-induced demagnetization of the sample magnets.

In relation to previous studies, this investigation went much further in terms of the range of absorbed doses to the irradiated sample magnets. Previous γ -ray irradiations of Nd-Fe-B sample magnets using a 60 Co source only went up to a maximum absorbed dose of 280

Mrad [5,7,9], whereas this study found no measurable radiation-induced demagnetization up to an irradiation dose of 700 Mrad.

X-ray Irradiation

The APS beamline 9 BM was used for the systematic x-ray irradiation of the Nd-Fe-B sample magnets. The sample magnets were placed in a specially designed holder inside the experimental hutch and irradiated with an x-ray spectrum with a peak energy of approximately 40 keV from the bending magnet source in Sector 9 of the storage ring. Seven sample magnets were irradiated to various absorbed doses, ranging from 30 Mrad to approximately 280 Mrad, during multiple trials.

The magnet irradiation configuration, placed inside the experimental hutch, consisted of a water-cooled magnet holder and a water-cooled aluminum spreader. The configuration was placed on a pedestal, which allowed for alignment of the sample magnet to the height of the beam window. Figure 5 provides a schematic diagram of the steel magnet holder with copper cooling tubes, and Figure 6 provides a schematic diagram of the aluminum spreader with copper cooling tubes. The aluminum spreader was placed directly after the beam window in the experimental hutch, between the window and the magnet holder. The purpose of the aluminum spreader was to diffuse the dose received by the sample magnet, allowing a more uniform irradiation of the magnet. A PHOTON® calculation showed that the 1-cm-thick spreader alleviated some of the heat load to the magnet by cutting the flux to the sample magnet from $4x10^{11}$ photons/second to $2x10^{10}$ photons/second [35]. The magnet holder was placed directly after the aluminum spreader. A photograph of the irradiation configuration is shown in Figure 7. During irradiation the sample magnet was wrapped in cardboard and placed in the center opening of the magnet holder, as shown in the photograph.

The temperature during the irradiation had to be kept below 50°C due to the temperature constraint of the radiachromic films that were used for dosimetry purposes. The temperature also had to be controlled due to the temperature sensitivity of the sample magnets themselves, in terms of temperature-induced demagnetization. Chilled water was circulated through copper cooling tubes to control the temperature of the aluminum spreader as well as the magnet holder. As a result of the chilled magnet holder, the temperature of the sample magnet was maintained at 45°C during irradiation, well below the 50°C requirement. A temperature measurement system comprised of a control program installed on a PC, and thermocouples, was used to monitor temperatures. Thermocouples were placed on the top and bottom of the aluminum spreader, on both sides of the sample magnet, and on the top of the magnet holder. A sixth thermocouple was used to monitor the ambient air temperature inside the experimental hutch. Prior to irradiation of the sample magnets, the temperature of the chilled water had to be adjusted to a level that would keep the sample magnet temperature well below 50°C during irradiation. A series of "dummy" magnet irradiations was done for this purpose. Stainless-steel "dummy" magnets were placed inside the magnet holder and irradiated while the temperatures were closely monitored. Numerous "dummy" irradiations were performed until the temperature of the water chiller was adjusted to a point that allowed for stabilized magnet temperatures below 50°C during irradiation. Figure 8 provides a sample of a temperature stabilization plot.

Along with temperature stabilization, a preliminary dose rate had to be determined prior to irradiation of the sample magnets. Radiachromic films were used to determine an approximate dose rate to the sample magnet, which was normalized, or based on the irradiation time and the beam current. Radiachromic films were placed on the top and the bottom of a sample permanent magnet, which was then placed inside the magnet holder and irradiated for a specified period of time. The average normalized dose rate was determined to be approximately 0.15 Mrad/hr/mA.

A PHOTON® calculation [35] was done to determine the bending magnet radiation spectrum that was used to irradiate the sample magnets at APS beamline 9 BM. The calculation also provided a calculated dose rate to the sample magnets that could be compared to the dose rate measured using the radiachromic films. The calculation was done for the specific parameters of APS beamline 9 BM, including the width, thickness, and composition of the windows that the beam passes through as it enters the experimental hutch. Figure 9 displays the calculated bending magnet irradiation spectrum for APS beamline 9 BM. The peak energy is approximately 40 keV with the 1-cm aluminum spreader in place. The calculated tissue dose to the magnets was determined to be approximately 36.5 Mrad/hr. Because only 1/3 of the beam is incident on the magnet, the tissue dose to the magnet is 1/3 of 36.5 Mrad/hr, or approximately 12 Mrad/hr. This is consistent with the normalized absorbed dose measured using the radiachromic films for an average beam current of approximately 80 mA.

Seven sample magnets were irradiated with x-rays at APS beamline 9 BM to various absorbed doses ranging from approximately 30 Mrad to 280 Mrad. Two of the sample magnets were irradiated using a "stepwise" irradiation procedure of three, three-hour irradiations, resulting in a total irradiation time of nine hours. The other sample magnets were irradiated as a result of one continuous irradiation. Table 8 provides a record of the irradiated sample magnets along with the associated absorbed doses. The absorbed doses to the sample magnets were determined using the 0.15-Mrad/hr/mA normalized dose rate approximation, due to the response limit of radiachromic films at dose levels greater than 10 Mrad. The 0.15-Mrad/hr/mA normalized dose rate approximation was determined by taking an average of normalized dose rates measured using radiachromic films.

Five measurements of the intrinsic remnant induction were taken for each sample magnet prior to irradiation. These measurements were also taken for the two control magnets. The average of these measurements served as initial readings to be compared to measurements taken after irradiation. Six radiachromic films were also prepared for the irradiation. These films provided an approximate absorbed dose to the magnet. Two of the films were placed on the top of the magnet and two were placed on the bottom of the magnet before the magnet was placed inside the holder. The other two radiachromic films served as controls. With the radiachromic films in place, a layer of cardboard was wrapped around the magnet, and the magnet was placed inside the holder. The water chiller was turned on inside the experimental hutch prior to irradiation to allow the

temperatures of the holder, spreader, and magnet to come down to acceptable levels. Temperatures were monitored during the entire irradiation and maintained below 45°C. A sample plot of the monitored temperatures is given in Figure 8.

Following irradiation for the specified period of time, the magnet was removed and allowed to cool to room temperature in the lab. The radiachromic films were removed and read to determine an approximate absorbed dose to the sample magnet. Intrinsic remnant induction measurements were then taken for the sample magnet and the control magnets. The average of these "final" readings was compared to the "initial" readings taken prior to irradiation, using the pooled t-test described previously, to determine if any measurable radiation-induced demagnetization had occurred. This process was then repeated for the next irradiation.

Results and Analysis of X-ray Irradiation

Intrinsic remnant induction measurements for each sample magnet prior to x-ray irradiation compared to measurements following irradiation revealed small, but not insignificant levels of variation. Tables 9-15 show the results as a percent change for each x-ray irradiation. The same results are displayed in a graphical form in Figure 10. This small, but not insignificant level of variation averaged around a 0.2% difference and was observed in approximately half of the x-ray irradiations performed.

As stated previously, a 0.5% change in the magnetic field of insertion devices typically cannot be tolerated [5]. Therefore, a 0.2% change would not be considered insignificant. The fact that the 0.2% variation only occurred in the sample magnets and not the control magnets also indicates a significant level of variation. However, a 0.2% level of variation is at the edge of the precision capabilities of the measurement device, or fluxmeter, rendering the observed variations statistically insignificant. In other words, the observed variations cannot be solely attributed to radiation-induced demagnetization due to the precision level of the fluxmeter and are determined statistically insignificant as a result.

The observed variations measured from the stepwise irradiations of Magnet 13A and Magnet 14A did not display a cumulative effect, suggesting that an annealing process was taking place between irradiations. However, continuous irradiation of the sample magnets did not reveal the existence of an annealing process. If an annealing process were responsible for the noncumulative effect observed during the stepwise irradiation procedure, we would expect to see larger changes in the intrinsic remnant induction of the sample magnets for longer continuous irradiations. The continuous irradiation of a sample magnet for twenty-four hours did not reveal any such changes in the intrinsic remnant induction, indicating that x-ray irradiation and an annealing process were not responsible for the observed variations in the intrinsic remnant induction measurements. The average 0.2% level of variation was observed during some of the stepwise irradiations, as well as during some of the continuous irradiations, over a range of irradiation times from six hours to seventeen hours. The noncumulative effect observed during the stepwise irradiation may be due to the precision capabilities of the instrument.

Statistical inference and the pooled *t*-test were used to compare the preirradiation means of the intrinsic remnant induction measurements to the means found after x-ray irradiation of the sample magnets. Many of the comparisons done using the test revealed evidence of differences between the means. However, the average 0.2% level of variation, or difference between the means, is at the edge of the precision capabilities of the fluxmeter. Therefore, although differences between the means may exist, they are probably attributable to the precision level of the instrument rather than radiation-induced demagnetization.

Previous to this study a pure x-ray irradiation of Nd-Fe-B sample magnets had never been done. Numerous studies involving the irradiation effects of direct electron beams on Nd-Fe-B permanent magnet samples have been conducted, but direct electron beam irradiation yields results for situations when the particle beam is lost directly on the magnet. With beam position monitors and feedback systems it is difficult to missteer a particle beam directly onto the permanent magnet structure at today's synchrotron light source facilities.

Radiation dose measurements of the insertion devices at the APS indicate the largest component of the absorbed dose to the devices is directed toward the downstream end of the device, likely the result of the greater amount of synchrotron radiation present at the downstream end [19]. With x-rays likely to be the largest absorbed dose component to the permanent magnets used in the APS insertion devices, pure x-ray irradiation, rather than direct electron beam irradiation, is a more realistic approach to simulate the irradiation of the permanent magnets under routine storage ring conditions.

Summary and Conclusions

The high remanence, or residual magnetic induction, and the intrinsic coercivity of Nd-Fe-B permanent magnets makes them desirable for use in insertion devices, but the high radiation environment within high-energy storage rings makes determination of the degree of radiation sensitivity, as well as the mechanisms responsible for radiationinduced demagnetization essential. For this study, sample Nd-Fe-B permanent magnets were irradiated up to an absorbed dose of 700 Mrad using 60Co γ-rays and up to an absorbed dose of approximately 280 Mrad using the APS bending magnet x-rays. In both cases a variation less than 0.2% was found to exist, indicating that radiation-induced demagnetization had occurred. The results of this study, along with previous investigations, indicate varying degrees of degradation in the intensity of magnetization of Nd-Fe-B permanent magnets based on the irradiation source, the total absorbed dose to the magnets, and the precision capabilities of the measuring device, to name a few of the variants. Results from previous investigations displayed a wide variation in the sensitivity to radiation, dependent on the manufacturing process or magnet vendor. Among the different Nd-Fe-B compounds, it was also found in previous studies that those with higher remanence and lower coercivity were more susceptible to demagnetization from an electron beam.

This study, along with previous studies, attempted to quantify the radiation-induced demagnetization of Nd-Fe-B permanent magnets from γ-rays and x-rays. Due to the many factors involved in the determination of radiation-induced demagnetization of Nd-Fe-B permanent magnets, along with great number of variations within each experimental procedure, it is difficult to make comparisons between investigations. The important point for each study is that a 0.5% change in the magnetic field of the insertion devices used in many synchrotron light sources typically cannot be tolerated. Therefore, even small variations of 0.1%-0.2% are important, as long as they can be determined statistically significant. For this study, the average 0.1%-0.2% level of variation was at the edge of the precision capabilities of the instrument and as a result could not be solely attributed to the radiation-induced demagnetization of the sample magnet. The variation was more likely explained by the precision capabilities of the instrument. In this case the 0.1%-0.2% levels of variation were determined statistically insignificant.

Although the results could not be determined statistically significant due to the precision capabilities of the instrument, this particular study did extend beyond the scope of previous investigations. Gamma irradiation of the permanent sample magnets using a ⁶⁰Co source was investigated up to an absorbed dose of 700 Mrad, and the first investigation of the radiation-induced demagnetization of Nd-Fe-B sample magnets using a pure x-ray source was conducted.

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Table 1: Magnet and Coil Parameters

Magnet Parameters:						
Average Dimensions						
	Length	5.01 cm				
	Width	4.75 cm				
	Thickness	0.675 cm				
Volume:						
	Length x Width x Thickness	16.06 cm ³				
Coil Paramete	rs:					
Coil Resistance		4.90 Ohms				
Helmholtz Constant	<u> </u>	0.161				
Diameter		8.0 inches				

Table 2: Results of 100-Mrad Gamma Irradiation

	Preirradiation		Postirra	diation (100 Mrad)	
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesl	a)	(Bdi-Tesl	a)	(%)
1A	2.544	5.477E-04	2.545	1.949E-03	0.039%
2A	2.544	5.477E-04	2.543	2.408E-03	0.039%
3A	2.557	5.477E-04	2.557	2.588E-03	0.000%
4A	2.546	5.477E-04	2.544	1.949E-03	0.079%
Control 1	2.553	8.367E-04	2.554	1.517E-03	0.039%
Control 2	2.551	5.477E-04	2.551	1.789E-03	0.000%

Table 3: Results of 200-Mrad Gamma Irradiation

	Preirradiation F		Postirradiation (200 Mrad)		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		(%)
1A	2.544	5.477E-04	2.539	1.414E-03	0.197%
2A	2.544	5.477E-04	2.540	1.581E-03	0.157%
3A	2.557	5.477E-04	2.552	2.121E-03	0.196%
4A	2.546	5.477E-04	2.541	1.342E-03	0.196%
Control 1	2.553	8.367E-04	2.548	1.643E-03	0.196%
Control 2	2.551	5.477E-04	2.547	1.100E-03	0.157%

Table 4: Results of 300-Mrad Gamma Irradiation

	Preirradiation F		Postirradiation (300 Mrad)		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		(%)
1A	2.544	5.477E-04	2.541	1.100E-03	0.118%
2A	2.544	5.477E-04	2.541	1.342E-03	0.118%
3A	2.557	5.477E-04	2.554	1.342E-03	0.117%
4A	2.546	5.477E-04	2.543	1.924E-03	0.118%
Control 1	2.553	8.367E-04	2.550	2.793E-03	0.118%
Control 2	2.551	5.477E-04	2.549	1.000E-03	0.078%

Table 5: Results of 400-Mrad Gamma Irradiation

	Preirradiation		Postirradiation (400 Mrad)		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		(%)
1A	2.544	5.477E-04	2.539	8.367E-04	0.197%
2A	2.544	5.477E-04	2.539	1.140E-03	0.197%
3A	2.557	5.477E-04	2.551	1.140E-03	0.235%
4A	2.546	5.477E-04	2.541	1.140E-03	0.196%
Control 1	2.553	8.367E-04	2.548	1.483E-03	0.196%
Control 2	2.551	5.477E-04	2.546	1.095E-03	0.196%

Table 6: Results of 500-Mrad Gamma Irradiation

	Preirradiation		Postirradiation (500 Mrad)		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		(%)
1A	2.544	5.477E-04	2.540	8.944E-04	0.157%
2A	2.544	5.477E-04	2.540	1.000E-03	0.157%
3A	2.557	5.477E-04	2.551	8.944E-04	0.235%
4A	2.546	5.477E-04	2.540	8.944E-04	0.236%
Control 1	2.553	8.367E-04	2.550	1.304E-03	0.118%
Control 2	2.551	5.477E-04	2.548	5.477E-04	0.118%

Table 7: Results of 700-Mrad Gamma Irradiation

	Preirr	adiation	Postirradiat	Postirradiation (700 Mrad)		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference	
	(Bdi-Tesla)		(Bdi-Tesla)		(%)	
1A	2.544	5.477E-04	2.541	1.095E-03	0.118%	
2A	2.544	5.477E-04	2.541	5.477E-04	0.118%	
3A	2.557	5.477E-04	2.554	1.304E-03	0.117%	
4A	2.546	5.477E-04	2.544	1.732E-03	0.079%	
Control 1	2.553	8.367E-04	2.551	1.949E-03	0.078%	
Control 2	2.551	5.477E-04	2.548	1.643E-03	0.118%	

Table 8: Sample Magnets Irradiated at APS Beamline 9 BM

Magnet	Irradiation Time	Absorbed Dose	Total Absorbed Dose
	(hours)	(Mrad)	(Mrad)
13A	3	35	35
	3 (6 total)	33	68
	3 (9 total)	41	109
14A	3	33	33
	3 (6 total)	42	75
	3 (9 total)	38	113
15A	9	117	117
16A	9	117	117
19A	16 hours, 40 minutes	197	197
20A	16 hours, 40 minutes	198	198
21A	24	280	280

Table 9: Results of 9-Hour Stepwise Irradiation of Magnet 13A

Magnet 13A: 3-hour irradiation

	Preir	radiation	Posti	rradiation	
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
13A	2.542	8.944E-04	2.541	1.140E-03	0.039%
17A: Control 1	2.549	1.225E-03	2.548	1.304E-03	0.039%
18A: Control 2	2.544	1.000E-03	2.543	1.304E-03	0.039%

Magnet 13A: 3-hour irradiation (6 total)

	Preir	radiation	Posti	rradiation	
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
13A	2.544	8.944E-04	2.537	8.367E-04	0.275%
17A: Control 1	2.549	1.643E-03	2.550	8.944E-04	0.039%
18A: Control 2	2.544	1.095E-03	2.544	8.944E-04	0.000%

Magnet 13A: 3-hour irradiation (9 total)

	Preirradiation		Posti	rradiation	
Magnet	Mean Standard Deviation		Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
13A	2.544	1.095E-03	2.540	7.071E-04	0.157%
17A: Control 1	2.550	8.944E-04	2.549	8.944E-04	0.039%
18A: Control 2	2.544	1.000E-03	2.545	8.944E-04	0.039%

Table 10: Results of 9-Hour Stepwise Irradiation of Magnet 14A

Magnet 14A: 3-hour irradiation

	Preir	radiation	Posti	rradiation	
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
14A	2.542	3.317E-03	2.536	8.367E-04	0.236%
17A: Control 1	2.548	2.000E-03	2.547	1.517E-03	0.039%
18A: Control 2	2.543	1.643E-03	2.543	8.944E-04	0.000%

Magnet 14A: 3-hour irradiation (6 total)

	Preirradiation		Postirradiation			
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference	
	(Bdi-Tesla)		(Bdi-Tesla)			
14A	2.537	5.477E-04	2.540	1.095E-03	0.118%	
17A: Control 1	2.546	1.414E-03	2.549	1.095E-03	0.118%	
18A: Control 2	2.543	8.944E-04	2.543	8.944E-04	0.000%	

Magnet 14A: 3-hour irradiation (9 total)

	Preirradiation		Postirradiation		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
14A	2.539	1.581E-03	2.540	1.140E-03	0.039%
17A: Control 1	2.549	1.414E-03	2.547	2.916E-03	0.078%
18A: Control 2	2.543	8.367E-04	2.544	1.817E-03	0.039%

Table 11: Results of 9-Hour Continuous Irradiation of Magnet 15A

	Preirradiation		Postirradiation		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
15A	2.548	1.140E-03	2.547	1.140E-03	0.039%
17A: Control 1	2.549	1.225E-03	2.550	1.517E-03	0.039%
18A: Control 2	2.545	1.000E-03	2.544	1.000E-03	0.039%

Table 12: Results of 9-Hour Continuous Irradiation of Magnet 16A

	Preirradiation		Postirradiation		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
16A	2.551	1.483E-03	2.544	1.517E-03	0.274%
17A: Control 1	2.550	8.367E-04	2.549	8.367E-04	0.039%
18A: Control 2	2.545	4.472E-04	2.544	8.367E-04	0.039%

Table 13: Results of 16-Hour, 40-Minute Continuous Irradiation of Magnet 19A

	Preirradiation		Postirradiation		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
19A	2.546	1.140E-03	2.543	1.095E-03	0.118%
23A: Control 1	2.555	8.367E-04	2.553	1.225E-03	0.078%
24A: Control 2	2.529	1.000E-03	2.528	1.095E-03	0.040%

Table 14: Results of 16-Hour, 40-Minute Continuous Irradiation of Magnet 20A

	Preirradiation		Postirradiation		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
20A	2.546	1.342E-03	2.545	5.477E-04	0.039%
23A: Control 1	2.555	8.367E-04	2.554	5.477E-04	0.039%
24A: Control 2	2.529	1.000E-03	2.527	2.168E-03	0.079%

Table 15: Results of 24-Hour Continuous Irradiation of Magnet 21A

	Preirradiation		Postirradiation		
Magnet	Mean	Standard Deviation	Mean	Standard Deviation	Percent Difference
	(Bdi-Tesla)		(Bdi-Tesla)		
21A	2.543	5.477E-04	2.543	4.472E-04	0.000%
23A: Control 1	2.556	8.367E-04	2.556	8.367E-04	0.000%
24A: Control 2	2.530	8.367E-04	2.531	8.367E-04	0.040%

R N-R

R N-R

R N-R

R N-R

R N-R

H H

Where
$$R = C - COH$$
H H

Figure 1: Radiation-Induced Photochemical Reaction

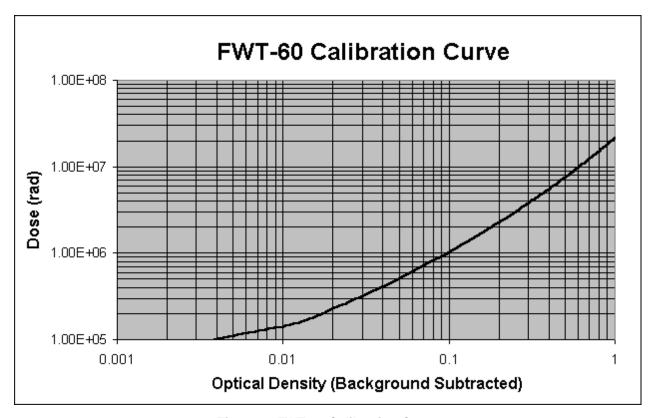


Figure 2: FWT-60 Calibration Curve

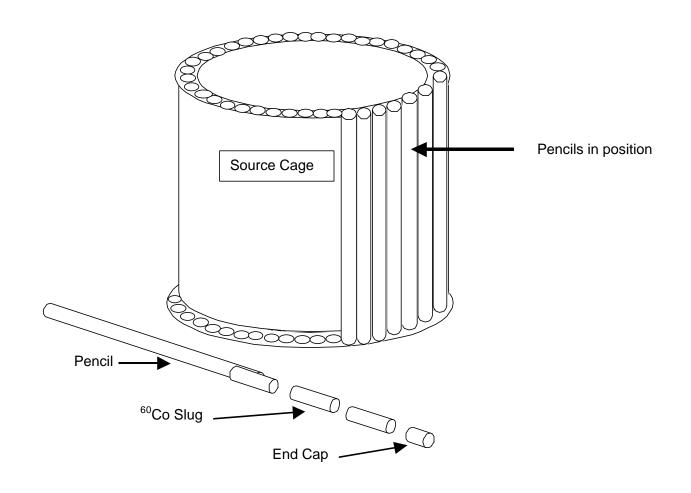


Figure 3: Source Cage and "Pencil" Source Arrangement

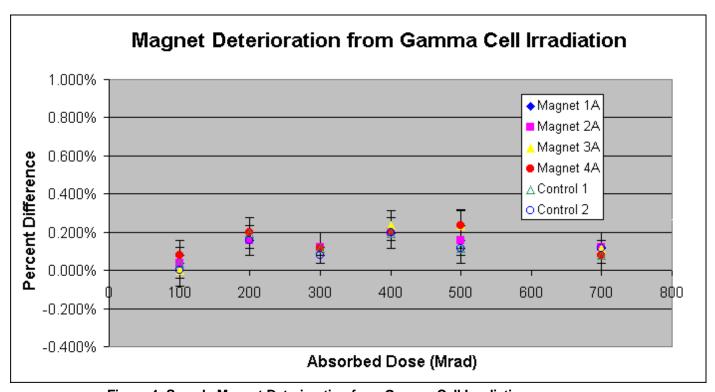


Figure 4: Sample Magnet Deterioration from Gamma Cell Irradiation

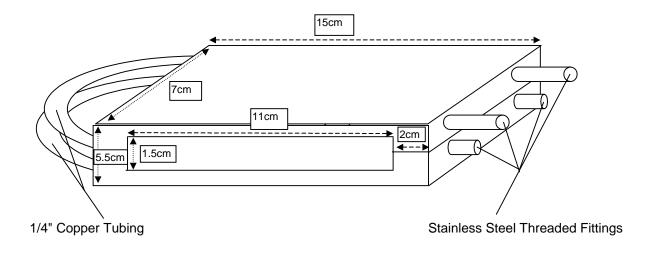


Figure 5: Steel Magnet Holder with Copper Cooling Tubes

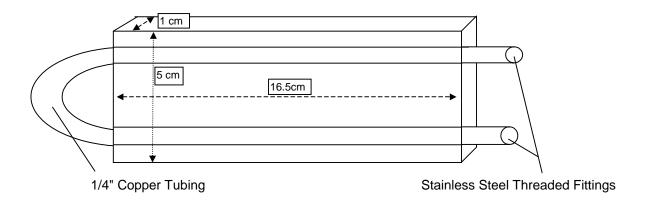


Figure 6: Aluminum Spreader with Copper Cooling Tubes

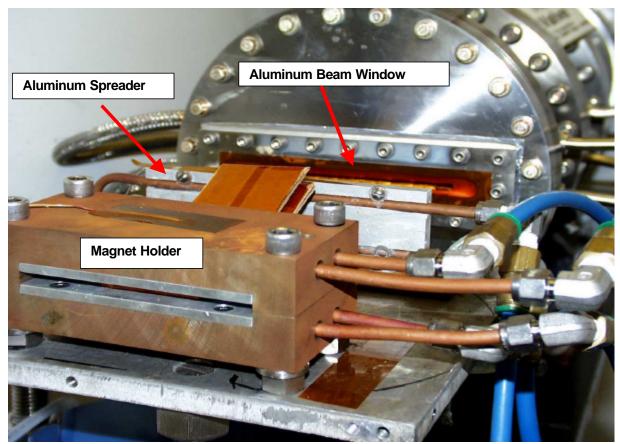


Figure 7: Sample Magnet Irradiation Configuration

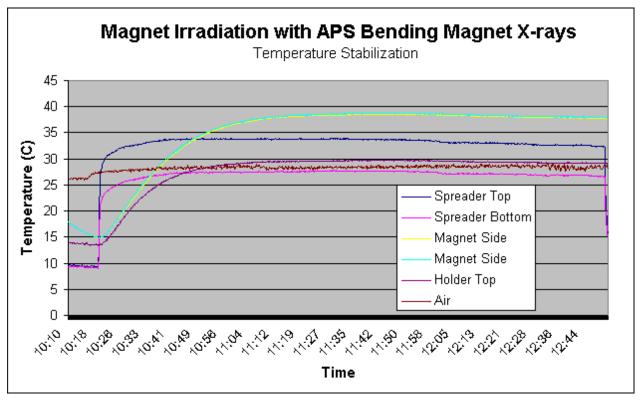


Figure 8: Temperature Stabilization Plot

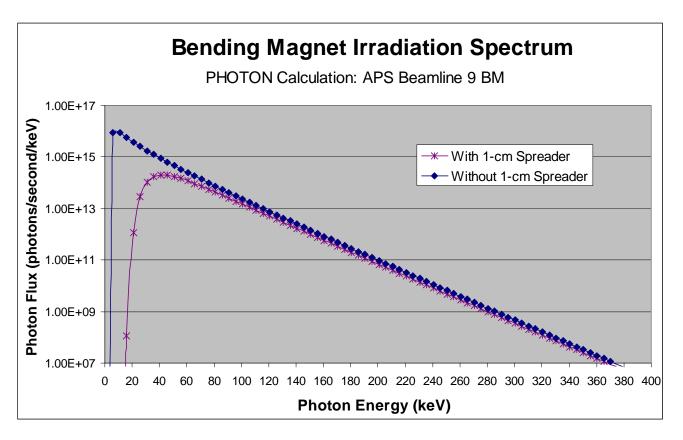


Figure 9: Calculated Bending Magnet Irradiation Spectrum for APS Beamline 9 BM

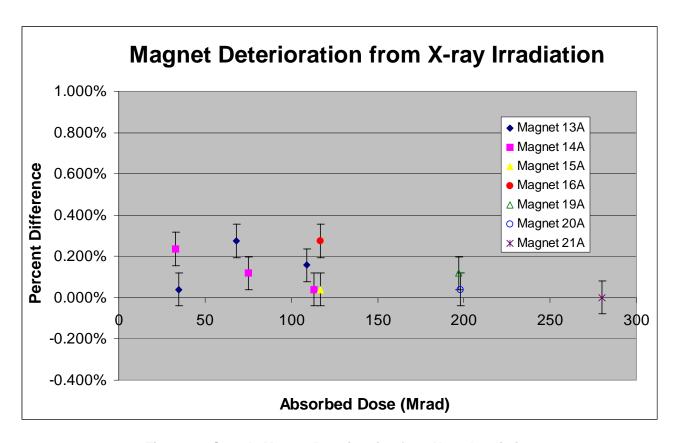


Figure 10: Sample Magnet Deterioration from X-ray Irradiation